APPENDIX

Changes to Claims:

The following are marked-up versions of the amended claims:

3. (Amended) The process according to claim 1 ~~or 2~~, wherein in step a) at least two product layers are applied on said partial surface of said carrier (5).

4. (Amended) The process according to claim 1 ~~any one of claims 1 to 3~~, wherein steps a) to c) are performed during one rotation of said carrier (5).

5. (Amended) The process according to claim 1 ~~any one of claims 1 to 3~~, wherein steps a) and b) are performed during at least two rotations of said carrier (5) and are followed by step c).

6. (Amended) The process according to claim 1 ~~any one of the preceding claims~~, wherein steps a), b) and c) are performed continuously and simultaneously on different partial surfaces of said carrier at a same angular velocity of said carrier.

7. (Amended) The process according to claim 1 ~~any one of the preceding claims~~, wherein a method of coating under vacuum is used in step a).

10. (Amended) Apparatus according to claim 8 ~~or 9~~, wherein said separating agent is an inorganic separating agent which may be evaporated in vacuum without dissociation, said product layers include metals, oxides, fluorides or carbides, and said carrier (5) comprises metal, glass, enamel, ceramic, or an organic material.

11. (Amended) The apparatus according to claim 7 ~~any one of claims 7 to 10~~, wherein said carrier (5) comprises an open or closed, rotationally symmetrical, rigid body.

12. (Amended) The apparatus according to claim 7 ~~any one of claims 7 to 10~~, wherein said carrier (5) comprises several open or closed, rotationally symmetrical, rigid bodies which rotate about a common axis or about several axes.

16. (Amended) The apparatus according to claim 8 ~~or 9~~, comprising means for coating said carrier with a separating agent layer prior to application of said product layer, wherein said separating agent is a meltable organic separating agent, said product layers include metals, oxides, fluorides or carbides, and said carrier (5) comprises metal, glass, enamel, ceramic, or an organic material.

Description

Process and Apparatus for Producing Plane-parallel Platelets

The present invention relates to a process for producing plane-parallel platelets and an apparatus for implementing this process.

Plane-parallel platelets are used as pigments in paints and printer's inks and are characterized over ground pigments by their capability of being produced at a very small thickness. As their planar surfaces align in parallel with the surface of the base following paint application, they generate a directed reflection of impinging light in comparison with ground pigments which reflect more or less diffusely. In the prior art, the manufacture of such plane-parallel platelets, which have uses far beyond the decorative range, is carried out substantially in accordance with three processes:

a) The coating of natural mica platelets with highly refractive layers, mostly of TiO_2, is effected by stirring the platelet in a titanium-containing solution and subsequently heating it in air to approx. 500 - 800°C. Products under the trade names IRIODIN® and AFFLAIR® are examples herefor. Coating methods are also known from the prior art and mostly take place in a fluidized bed heated to 400 - 600°C, where the reaction $TiCl_4 + 2\ H_2O \rightarrow TiO_2 + 4\ HCl$ is employed. Organic titanium compounds are furthermore used, such as iron or cobalt halides, or carbonyls.

b) In order to not have to rely on the natural substance mica, processes were developed as are described,

for example, in WO 93/08237. Here a thin layer of a liquid, silicate-containing substance is applied on a circulating belt in air. The substance is afterwards dried on the belt, reacts to SiO_2 in a subsequent acid bath, is then washed in another water bath, and is afterwards scraped off the belt. The thin quartz platelets obtained after an annealing step are chemically coated with further oxides in another process in the fluidic bed or stirring process. A survey of such chemical coatings is described by Schmid and Mronga in "Luster Pigments with Optically Variable Properties" (Report by the European Coatings Conference, April 7 - 9, 1997, Nuremberg)

c) In order to produce plane-parallel platelets having a controlled thickness, the vapor deposition (PVD = physical vapor deposition) method has been used for years. Examples herefor are listed in the patent specifications U.S. 3,438,796 of 1967 to Dupont and U.S. 5,135,812 to Flex Products, wherein a Fabry-Pérot reflex filter is produced as a pigment. The product exhibits a strong color change with a changing angle of light incidence and is printed on bank notes as a protection against counterfeiting. In these manufacturing processes, a polyester sheet coated in advance with a paint serving as a separating agent in accordance with a known method is used as a carrier. Subsequently, the layer system is applied on the carrier by repeated vapor deposition in vacuum of the various layers. The sheet roll is taken from the vacuum chamber and on another machine passes through a bath in which the paint layer is dissolved in a suitable solvent. The product then falls off the carrier as coarse flakes which are further processed by separating the solvent, drying, grinding. The sheet carrier can be used once only and thus incurs considerable costs. In patent specification U.S. 3,123,489 to Bolomey, the use of a carrier is disclosed on which a large alternating layer sequence of a salt as a separating

agent and zinc sulfide as a product are vapor deposited. The carrier here is a circulating belt or a rotary table of a known type as used in optical vapor deposition. After a large number of alternating coatings with separating agent and product, the carrier is taken from the apparatus and watered, with the salt layers between the product layers dissolving in the process, and the product being present in the form of small platelets in a suspension. This material, mostly zinc sulfide, is used in cosmetics and for decorative purposes as a synthetic pearl luster. Notwithstanding the simple construction of the vacuum apparatus, it is a drawback that it is not a continuous process and that the vapor deposited layers, overlying each other in large numbers, at increasing thickness form columnar structures exhibiting but a diffuse reflexion. This effect is desired in the case of pearl luster, however not with pigments used as paint on automobile bodies or as metallic inks.

A German patent application not laid open yet at the time of this application relates to use of a circulating metal belt on which the separating agent and the sequence of layers of the plane-parallel platelets to be produced are successively vapor deposited in a high vacuum. Afterwards the metal belt passes through another vacuum chamber having a higher pressure, wherein the separating agent is dissolved in a liquid, usually water at a temperature of 35°. The use of many locks, which would be raised from the high vacuum to atmospheric pressure by the metal belt, is hereby avoided. Water at 35°C has a vapor pressure of 54 mbar only. Additional locks employed between 54 and 1000 mbar are omitted. The process entirely takes place under vacuum between about 10^{-4} and 54 mbar. Only the product having the form of a suspension is taken to atmospheric pressure. Despite this progressive technique, nevertheless a belt material is still needed which is

subjected to mechanical strain on deflection rollers. The fatigue strength limit of the belt is exceeded after a certain number of cycles, which accordingly has to be exchanged.

The simultaneous action of a salt solution further reduces the fatigue strength, typically by a factor of 2-3 (Thyssen Edelstahl Techn. Ber. 7/1981, vol. 1, pp. 68-69), so that it is necessary to exchange the metal belt in certain intervals. The use of highly polished carrier surfaces having a very low surface roughness, such as glass, quartz, glazed ceramic or enamelled steel, is not possible in the case of a belt running over indispensable deflection rollers.

It is an object of the present invention to propose a process wherein the production of plane-parallel platelets is carried out effectively, independent of the kind of the carrier material used. During the entire production process the vapor deposited layers should not contact any other surfaces until the product is stripped from the carrier. To be more precise, layers, in particular layers for infrared applications and microwave absorbers, which may consist of up to 35 single layers, should not prematurely flake off the carrier due to flexure on deflection rollers.

This object is attained through a process according to claim 1 and an apparatus according to claim 8.

Through provision of both coating means and stripping means on a carrier rotatable about an axis, the partial surface of which between these two means may be transported by rotating the carrier, a product layer may be applied and removed continuously, and thus the production of plane-parallel platelets may be performed efficiently and with low waste.

Preferably before the product layer a separating agent layer is applied, whereby easy stripping of the product layer by dissolving the separating agent is made possible.

The product layer may also be made to have a plurality of layers, whereby multiple-layer platelets having more complex properties may be produced. Here it is possible to apply the product layer during one rotation or several rotations of the carrier. This allows for variable adaptation of the conditions to the various properties of product layers.

Through at least two revolutions of the carrier prior to stripping the multiple layers as a packet, the liquid in the stripping means is discharged, so that no stripping will take place prior to completion of the packet of layers.

The separating agent is evaporated at a vacuum providing a sufficiently long Mean Free Path for the separating agent molecules. In adaptation to the geometrical conditions of a coating installation, the Mean Free Path is to be about 10 to 50 cm. This corresponds to a required vacuum in the evaporation chamber of approximately 1×10^{-4} to 5×10^{-4} mbar.

It is possible to use any separating agent, with inorganic separating agents having better suitability, particularly when layers having great thicknesses of >5 microns or layers at very high temperatures are to be vapor deposited. The high thermal load of the carrier then precludes the use of organic separating agents. Examples for evaporation substances are chromium, titanium, nickel, oxides of aluminum, titanium and silicon.

When using an organic separating agent, it is advantageous if, for stripping the product layer, the carrier immerges into the organic substance where the separating agent layer is then melted.

It is moreover advantageous if different partial surfaces are located under the various means at a same point of time, so that one partial surface will be coated while a product layer is simultaneously being removed from another partial surface. An efficient operation of such an apparatus is thereby made possible.

Through the use of coating processes under vacuum an efficient process flow and high quality of the platelets may be attained. Here the separation between ranges of different pressures is of importance. As coating processes, it is possible to employ, for example, vapor deposition, sputtering, plasma polymerization, or a combination of these processes in the same vacuum chamber under vacuum.

As the carrier, a multiplicity of rotationally symmetrical bodies may be employed, whereby an adaptation to the properties of the platelets as desired by the customer is possible at low expense.

A face-and-back coating of the carrier or/and a parallel coating of several carriers also contributes to increasing the yield of platelets.

Developments in accordance with the invention constitute the subject matters of the subclaims.

Hereinbelow the invention will be explained by referring to the annexed drawings, wherein

Fig. 1 and Fig. 2 show front and lateral views of the apparatus according to the invention,

Fig. 3 shows a first modification of the apparatus according to the invention,

Fig. 4 shows a second modification of the apparatus according to the invention,

Fig. 5 shows a third modification of the apparatus according to the invention, and

Fig. 6 illustrates the functional principle of the stripping step.

In order to produce plane-parallel platelets according to the invention of metals, oxides, fluorides, nitrides and carbides and other substances applicable in vacuum in arbitrary combinations, the apparatus is essentially composed of the following elements as shown in Figs. 1 and 2:

A vacuum chamber 100 having an intermediate separation 1 which includes two connecting openings 2a and 2b and divides the vacuum chamber 100 into an evaporation part 3 and a product collection part 4.

A rotating, rigid carrier 5 of metal, glass or enamelled steel or some other material having a surface as smooth as possible, and which may be coated under the prevailing vacuum conditions in vacuum by vapor deposition, sputtering or by means of a PECVD process. The carrier 5 is mounted centrically on a rotating shaft 6 rotatingly driven by a drive mechanism 7 arranged outside the vacuum chamber. In the course of rotation, each sector of the rotating, rigid carrier 5 is moved past the evaporator 8 for

separating agent, the evaporator(s) 9a, 9b, 9c determining the sequence of layers of the product.

The evaporators 9a, 9b, 9c have a known design such that evaporation material, which is present in the form of wire, plate metal or granules, will be supplied continuously. For maintenance purposes, the evaporators 9a, 9b, 9c may be withdrawn from the work zone into another vacuum chamber 11 capable of being closed with the aid of a lock 10 having a generally known design, and may there cool down under vacuum.

Separating agents suited for evaporation in vacuum are in a known manner chlorides, borates, fluorides, hydroxides and other inorganic substances. Several are described in the patent specifications U.S. 5,156,720 to Rosenfeld and Smits and U.S. 3,123,489 to Bolomey.

In order to achieve identical layer thicknesses, measurement of each single layer is carried out by optical measurement of the reflected light where the layers are transparent. On a metallic substrate, transparent layers exhibit different interference colors depending on their layer thickness n × d (n: refractive index of the layer material, d: geometrical thickness). These colors may be used for regulating the desired layer thickness through spectral measurement of the reflected light. Apart from this, there exists in accordance with DE 4338907 a process which measures the thickness increase owing to metal condensate on a wire passing through the vapor jet by means of laser thickness measurement, and continuously performs mathematical conversion to the layer thickness applied on the carrier.

Having passed through the evaporator or sputter zones, the rotating, rigid carrier 5 passes through a narrow

passage 12a and 12b in the intermediate separation 1. The passages 12a and 12b are designed such that the walls thereof maintain a constant distance, typically of 0.5 - 1 mm, from the rotating, rigid carrier. The like distance control is part of the prior art and provides for a low gas flow to the evaporation part 3. Having passed through the passage 12a, the coated portion of the carrier 5 immerges into a bath 13 arranged underneath the intermediate separation 1 and accommodating a liquid that a) has a low vapor pressure and b) constitutes a good solvent for the vapor deposited separating agent.

Such liquids are: secondary and tertiary alcohols such as ethylene glycol, propylene glycol, glycerol and their derivatives, but also higher primary alcohols and their derivatives. In the temperature range of technical interest of 20 - 50°C, these liquids have a saturation vapor pressure of 0.01 to 0.05 mbar, whereas in the evaporation part 3 a vacuum of typically 3×10^{-4} mbar is generated. The gas flow rate through the connecting openings 2a and 2b only in the intermediate separation 1 would be too high to be able to maintain a pressure difference of 0.05 mbar at the intermediate separation 1 at sensible pumping expense. The gas flow rate through thin split tubes 14a, 14b, in accordance with Wutz, Theorie and Praxis der Vakuumtechnik, ISBN 3-528-04884-0, page 101, Gl. 4.95 is reduced by a factor 25.3 if, in the place of a connecting opening covering a distance of 0.2 cm, a split tube having a length of 20 cm is used between the product collection part 4 and the evaporation part 3. By replacing the gaps in the intermediate separation with split tubes mounted there, which leave passages 12a, 12b of 2 × 0.05 cm each and by separately and continuously pumping these passages to 10 - 2 mbar, the total gas flow is reduced to 0.11 mbar liter/sec for two passages having a with of 250 cm and a thickness of 2 × 0.05 cm each at a passage length of 20 cm

passages. This gas flow only causes a low strain on the high vacuum pumps 16. The product collection part 4 and the passages 12a, 12b are pumped by mechanical pumps 17, each comprised of a combination of mechanical vacuum pumps and rotary piston fans. The design of all vacuum pumps as to dimensions is dependent on the selected size of the vacuum chamber 100 and the working conditions. The technical literature knows numerous design methods in this respect.

In the further course, the separating agent layer 71 on the rotating, rigid carrier 5 vapor deposited with separating agent and with the product layers, is dissolved below the liquid level in the vessel 18 in accordance with Fig. 6 while mechanically aided in accordance with known methods. The non-soluble product layers 73 fall off the carrier 72 as small, flake-like particles. In subsequent processes, comminution of the plane-parallel platelets to the desired dimensions takes place later on. To this end, known comminution and classification processes are available, such as grinding and air separation at atmospheric pressure. As the last step, further processing into paints or inks takes place.

After completed stripping of the layer system, the suspension is conveyed from the vessel 18 to atmosphere in accordance with Fig. 1 by means of a liquid pump 15. The suspension flows through a filter array 19 or a centrifuge array of a known design which are located outside the vacuum chamber 100. From there, the liquid freed from particles returns into the vessel 18 after again having been taken to the working temperature of the vessel 18 in a heater 20.

In the further course, the carrier 5 again emerges from the liquid in the vessel 18. Residual traces of liquid are roughly removed by a scraper 21 and run back into the

vessel 18. A furthermore remaining film evaporates against a low-temperature surface 22 to condense there. After passing through a split tube 14b, the corresponding sector of the rotating, rigid carrier 5 is again located in the evaporator part 3. The circuit is closed.

A plant for producing platelet-shaped pigments in a PVD process in the presently described arrangement requires evaporators 9a, 9b, 9c capable of discharging vapor in a horizontal direction in long-term operation. Such evaporators are disclosed in the literature DE 4342574 (Weinert). Other evaporator versions evaporating in a horizontal direction are described in patent specification U.S. 2,665,226 (Godley).

The present invention relates not only to the described arrangement of Figs. 1 and 2, but also permits the following exemplary embodiments of Figs. 3 to 6, as well as other ones that are readily apparent to the person having skill in the art from the present disclosure.

Instead of producing smooth, planar platelets, however, plane-parallel platelets may also have a surface structure, for vacuum coating methods do not produce differences of thickness in the microscopic range. It is accordingly possible to produce desired surface patterns on the carrier in advance by previous etching in accordance with the photoresist method. Hereby it is possible to produce, instead of planar platelets, miniature reflectors focusing impinging light only a few millimeters in front of their surface thanks to their spherical shape. It is, however, also possible to produce platelets with optical gridlines or with sharp-edged, raised webs serving as predetermined breaking points for the production of platelets having a defined shape and size. Upon condensation from the vapor phase, an impression of the carrier's structure is

engendered on the carrier. Such patterns should be situated in the range of the particle size used later on, of about 520 microns. This is, however, under the precondition that such layers are stripped from the carrier 5 at each revolution so as not to lose their fine structure. This operating mode cannot be realized in known processes employing either a circulating metal belt or the vapor deposition of a large number of alternating separating agent and product layers.

In order to be able to employ electron beam evaporators 31 which only evaporate in a vertical direction upward from a bath surface 32, it is necessary, in accordance with Fig. 3, to rotate the carrier 33 about a vertical axis 34. Furthermore the vessel 35 needs to be adapted correspondingly. The carrier here does not immerge into the vessel 35. Instead, the layers are removed by brush-type, rotating members 36.

It is possible in accordance with Fig. 4 to attach evaporators 43a, 43b, 43c on either side of the rotating carrier 42.

It is equally possible in accordance with Fig. 5 to simultaneously operate several carriers 51a, 51b, in this case discs, on a same axis 52, wherein the number of the evaporators to be used is multiplied to one evaporator each for each carrier surface to be coated.

Instead of one or several disc-shaped carrier(s) or of one or several rotating rings having a concentric arrangement, it is possible to use cylinders having a horizontal axis. Any other rotationally symmetrical bodies may be used as carriers.

In order to produce plane-parallel platelets of a large number of repeated layers of the type (A,B)N or (A,B,C)N, wherein N is the number of repetitions of the combination of layers, and A,B,C are the evaporated substances, the following option is available without modification of the equipment:

a) The separating agent only is vapor deposited in the first step on the carriers, with all other evaporators having been blocked by a closable shutter, and the liquid in the vessel 18 in accordance with Fig. 1 does not get into contact with the carrier. This is realized by lowering the level inside the vessel 18.

b) Layers A and B or A,B,C are applied from two or three evaporators arranged on the path of rotation of the carrier 5, at a controlled layer thickness. After N rotations, the layer compound A,B or A,B,C will be present N times. The separating agent evaporator is here blocked by means of a shutter.

c) The evaporators having produced the layers A,B or A,B,C are blocked by means of shutters. As the rotation of the carrier 5 is continued, the liquid level in the vessel 18 is raised far enough to contact the vapor deposited layers. Owing to the microscopic cracks present in the layers, inherently brought about by tensions in the layers, the liquid will reach the separating agent even in the presence of a very large number of layers, and dissolves the separating agent, so that the product layers detach as flakes to then be present in the form of a suspension. The time required for this purpose, although longer than for products having a smaller number of single layers, nevertheless is less than 30 seconds.

d) The process is repeated analogously to a).

Such layer systems are known from the production of so-called metal oxide vaporized mirrors and infrared-reflecting surfaces alternatingly requiring up to 31 layers of a transparent material having a high refractive index on the one hand and a low refractive index on the other hand. In this case, the very layer system is the product which is present as small platelets having the desired optical properties, without having to use a carrier such as a glass plate or a glass reflector. It is furthermore advantageous that such platelets may also be produced to have structured surfaces, such as concave or convex micro-reflectors, and in comparison with layers deposited on a glass substrate remain transparent over a wide infrared range. The arrangement of the layer thicknesses is carried out in accordance with known principles of thin-layer optics. When using titanium oxide and magnesium fluoride, but also with combinations of other materials such as tantalum oxide, zirconium oxide, cerium oxide, zinc sulfide and other known substances for the highly refractive layers, and quartz or other fluorides as the low-refraction layer, there results a platelet material largely free of absorption in the range from 400 to 10,000 nanometers.

In the following, examples for the process flow for producing plane-parallel platelets of aluminum will be given.

<u>Example I:</u>

A vacuum chamber in accordance with Fig. 1 and Fig. 2 contains 2 evaporators arranged on a circular arc in the direction of rotation of the carrier. The first evaporator is filled with sodium tetraborate having had constitutional water removed in advance by annealing at 600°C in atmosphere, and is heated to approximately 1300°C at a

chamber vacuum of 2×10^{-4} mbar, while the carrier already is rotating about its horizontal axis. Temporally offset, the second evaporator for aluminum is heated to about 1500°C, and aluminum in wire form is fed in a known manner. In order to obtain the desired horizontal evaporation direction towards the carrier, a U-shaped, heated screen is arranged around the aluminum evaporator, preferably directing the metal vapor towards the carrier. Both evaporators are continuously operated until their supply of evaporator material is exhausted. In the meantime, the rotating carrier transports the layers present on it through a split tube-type passage into a chamber having a higher pressure of typically 0.04 mbar, where the coated portion of the carrier in the liquid immerges into a vessel wherein the aluminum layer is torn open, aided by ultrasound acting on the carrier in the liquid. The acting glycerol, having a vapor pressure of less than 0.04 mbar at its operating temperature of 50°C, rapidly dissolves the separating agent layer of sodium tetraborate.

The aluminum is now present in the liquid in the form of platelets. While the rotating carrier incessantly supplies new layers of sodium tetraborate and aluminum, the suspension is continuously sucked from the vessel by means of a liquid pump, is taken to a pressure of approximately 1.5 - 6 bar, and introduced into a jacketed centrifuge in atmosphere. Due to the difference in density, the aluminum platelets deposit on its wall, the clear liquid is discharged from the jacket, and via a valve flows back into the vessel placed under vacuum without inducing air. A heat changer in this glycerol cycle ensures that the temperature may be kept constant. The rotating carrier, now freed from layers, emerges from the liquid, initially passes through several mechanical strippers, and then moves between two panels that are kept at a very small distance from its surface and cooled to -30°C. As a result of the partial

pressure gradient, the glycerol still adhering to the carrier as a film of less than 1 micron evaporates and condenses on the cold surface, from which it runs off. On its further way, the carrier passes through a second split tube-shaped passage and again gets into the range of the separating agent evaporator with sodium tetraborate. The rotating carrier here consists of a highly polished metal plate of 3-mm stainless steel, the diameter of which is somewhat smaller than the diameter of the vacuum chamber. In the example, a vacuum chamber having a diameter of 2 m and a disc size of 1.9 m are used. Evaporators for separating agent and aluminum are installed on either side of the disc. The vapor deposited circular ring on the disc has a width of 0.60 m and rotates at a rate of 10 rpm. Per minute, 49 m² of aluminum platelets are obtained. The selected thickness of vapor deposition, depending on the use of the product, is 50 to 500 nanometers.

Example II:

In the vacuum chamber used in Example I, the rotational axis of the disc-shaped carrier having a diameter of 1.90 m is installed vertically. According to Fig. 3, one separating agent evaporator with anhydrous sodium chloride and three electron beam evaporators are located underneath the disc at its periphery. All evaporators are arranged on a same radius of the carrier. In a form modified in comparison with Example I, a layer of sodium chloride - aluminum oxide - titanium aluminum oxide is successively vapor deposited on the horizontal carrier at a vacuum of 7 x 10^{-5} mbar, wherein all the materials are continuously or discontinuously supplied to the evaporators. Corresponding means are known from literature. The stripping station following on the rotation path is different in that the edge of the vessel in which stripping of the layers takes place, is located very closely by the underside of the

rotating disc, however without contacting it. The vacuum in the space above the liquid level and the disc-shaped carrier is 0.04 mbar. Rotating brushes of a material suited for vacuum technology convey the liquid, in this case ethylene glycol at 30°C, to the carrier, and dissolve the separating agent. The product layers comprised of 3 layers become suspended in the liquid in the form of flakes. A dipping process is here not possible owing to the horizontal arrangement of the disc. The carrier rotates at a rate of 5 rpm. The obtained product has a thickness of 150 nanometers. The platelets are obtained in the same manner as explained in Example I.

<u>Example III:</u>

In the same arrangement of the carrier, the evaporator, and the stripping station of Example I, plane-parallel platelets of a particular number of recurring layers of the A,B or A,B,C type are to be produced. The layer system vapor deposited in this example consists of a separating agent layer, here: calcium chloride, and a sequence of layers of titanium oxide and magnesium fluoride, which is repeated 15 times and terminates with another titanium oxide layer. The essential difference from Examples I and II is that vapor deposition of the layers is performed according to the following sequence:

- The brushes stripping the product from the carrier are lowered sufficiently far so as to not be in contact with the carrier any more.

- The separating agent evaporator applies calcium chloride during one to two full revolutions of the carrier. Subsequently a shutter is inserted between it and the carrier in a known manner. Further separating agent does not reach the carrier any more.

- One evaporator each, filled with titanium oxide and magnesium fluoride, apply at controlled layer thicknesses pro revolution, at concurrent measurement of the applied layer thicknesses in accordance with known methods, a highly refractive layer and a low-refraction layer. After the desired number of layers has been reached, both evaporators are closed with a shutter.

- The rotating brushes, which have the task of removing the layer system from the carrier by means of a liquid, here by means of ethylene glycol, by dissolving the vapor deposited separating agent, are mechanically adjusted so as to get into contact with the carrier. Stripping and further processing of the platelets is performed in the manner described in Example I. The rotational velocity of the carrier is reduced to such an extent that stripping may take place in a single run.

- The rotating brushes are again adjusted so that they do not contact the rotating carrier.

- Repetition of the process begins.

It is advantageous that by this variant according to Example III, plane-parallel platelets of a large number of recurring pairs of layers or triple layers may be produced with few evaporators without interrupting the vacuum.

In Examples I to III, very small traces of the stripping liquid still get into the evaporation part in the form of a film of a few strata of molecules adhering to the carrier. This effect, which would be very detrimental in all other vacuum coating processes, here is even found to be useful in most applications because it reduces the adhesive strength of the vapor deposited separating agent

on the carrier and allows for more rapid and complete stripping in the liquid. As the vapor deposited layers themselves constitute the product, good adhesion to a surface is not being demanded.

All variants satisfy the concept of the invention which is based on the circumstance that a separating agent and the product layers are successively applied in vacuum on one or several rigid carriers rotating in a vacuum chamber with 2 or 3 vacuum zones during one rotation through 360° of the carrier, which is a rotationally symmetrical structure. These product layers are simultaneously stripped from the carrier in another location, and the suspension thereby obtained is removed from the vacuum chamber. Further processing of the suspension into the final products, such as metallically reflecting paints or printer's inks is carried out later on at atmospheric pressure by means of filtering, centrifuging, further comminution and introduction into a liquid carrier, which may be a paint or an ink.

A continuous layer system produced by means of a vacuum coating process on one or several movable, rotationally symmetrical, rigid carrier(s) thus includes one or several inorganic separating agent layers and product layers of metals, oxides, fluorides, nitrides or carbides deposited thereon, which are continuously removed from the carrier in a single revolution through the action of a liquid which dissolves the inorganic separating agent layer and at the same time strips the product layers that are not soluble in the liquid from the carrier while causing them to decompose into flakes. A rigid carrier rotating about an axis is located inside a vacuum chamber subdivided into at least two pressure stages by intermediate separations, wherein the range in which the rotating, rigid carrier crosses through the intermediate separations has the form of a

passage. In a first zone, the rotating, rigid carrier is coated with a separating agent which is soluble in a liquid and evaporable in vacuum without decomposition, and during the same rotation passes through a second zone including one or several evaporators which produce the product layers. The rigid carrier passes on its rotation path a third zone into a separately pumped, further vacuum chamber of 10^{-3} to 1 mbar, in which the separating agent layer or the separating agent layers is/are dissolved by means of a liquid having a vapor pressure lower than or equal to the pressure in the second vacuum chamber, wherein the insoluble product layer or the product layers detach from the carrier in the form of non-coherent particles to then be present as a suspension in the liquid. The rotating, rigid carrier is on its further rotation path withdrawn from action of the liquid, freed from residues of the liquid, and again supplied to the process of coating with separating agent and product layers.

The steps of vapor deposition with separating agent and with product layers, and dissolution of the separating agent in a liquid, take place at a same angular velocity of the rigid, rotating carrier continuously and simultaneously on different locations of the carrier.

By repeatedly arranging an evaporator for the separating agent and the evaporator for the product layers, several strata of product layers may simultaneously be produced during a same rotation of the rigid carrier.

The rotating, rigid carrier may consist of one or several parallel discs, one or several closed or open cylinders, or one or several closed or open, further rotationally symmetrical bodies rotating either on a common axis or on several axes.

If a rotating, rigid carrier comprised of several parallel discs is used, all or some of these may be coated face-and-back at the same time.

Besides the vapor deposition method, other methods of coating under vacuum such as sputtering or plasma polymerization, or a combination of these processes, may be used in the same vacuum chamber.

The rotating, rigid carrier has a surface consisting of metal, glass, enamel, a ceramic or an organic material, wherein the materials for the surface and of the carrier may be different.

The rotating, rigid carrier has a surface which may be either left in its natural form, polished, or structured.

The steps of coating with separating agent, coating - repeated at least once - with at least two different substances jointly making up a layer system, and stripping of this layer system take place successively and without interruption of the vacuum.

As an alternative for coating with an inorganic separating agent, coating may be performed with an organic separating agent. Thus there is the possibility, instead of dissolving the separating agent in a solvent, of applying an organic separating agent such as, e.g., wax, resin or a thermoplastic material for example by evaporation or in the liquid state by dipping, rolling, pouring or spraying onto the carrier. Examples for the organic separating agent are sodium, lithium, magnesium, aluminum stearate, fatty alcohols and wax alcohols of the type C_xH_yO, with $15 < C < 30$, paraffin waxes, branched and linear fatty acids with $C > 15$, and thermoplasts.

On the organic separating agent applied in this manner, at least one product layer may be vapor deposited in the same manner as in the case of the inorganic separating agent.

The organic separating agent is selected in accordance with four criteria:

- The separating agent must still be present as a solid body under the thermal load on the carrier owing to vapor deposition of the product layer.
- The separating agent must decompose in a very small degree only in the liquid molten state under vacuum and should have a minimum vapor pressure at the melting point, preferably of less than 1 mbar.
- It must not enter a chemical reaction with the product layer vapor deposited thereon.
- The separating agent used must be readily separable in the subsequent further processing of the pigments into paints or inks by known processes, or else be acceptable in the final product.

Upon further rotation of the carrier, the latter immerges in a bath having the same molten organic substance as the separating agent. When the organic substance dissolves there, the product layer falls apart into small particles which are then present in the organic substance as a suspension or sediment. In the further steps this suspension may in the same way be pumped off, filtered and supplied to further processing into paints or inks.

One advantage of using such a process with an organic separating agent resides in the fact that after emerging from the bath, a thin layer of the organic substance remains on the carrier, which then is again used as separating agent. Few pigment particles possibly still embedded in this separating agent layer are not of

importance and will pass through the process one more time without being subjected to further vapor deposition inasmuch as they are embedded in the separating agent.

The present invention thus relates to an apparatus for producing plane-parallel platelets including a carrier rotatable about an axis, means for coating a partial surface of the carrier with at least one product layer, means for stripping the product layer from the partial surface of the carrier in such a way that plane-parallel platelets are produced, with transport of the partial surface between the coating means and the stripping means taking place through rotation of the carrier. Means for coating the carrier with a separating agent layer prior to application of the product layer may be provided. In the stripping means, the separating agent layer is dissolved, and the plane-parallel platelets are released.

Claims

1. A process for producing plane-parallel platelets, comprising the steps of:

a) coating a partial surface of a carrier rotatable about an axis (5) with at least one product layer,

b) transporting said partial surface through rotation of said carrier (5) subsequently to step a),

c) stripping said product layer from said partial surface of said carrier subsequently to step b), in such a way that plane-parallel platelets are produced.

2. The process according to claim 1, wherein said partial surface of said carrier (5) is coated with an inorganic separating agent in step a) prior to application of said product layer, and said separating agent layer is dissolved in step c).

3. The process according to claim 1 or 2, wherein in step a) at least two product layers are applied on said partial surface of said carrier (5).

4. The process according to any one of claims 1 to 3, wherein steps a) to c) are performed during one rotation of said carrier (5).

5. The process according to any one of claims 1 to 3, wherein steps a) and b) are performed during at least two rotations of said carrier (5) and are followed by step c).

6. The process according to any one of the preceding claims, wherein steps a), b) and c) are performed continuously and simultaneously on different partial surfaces of said carrier at a same angular velocity of said carrier.

7. The process according to any one of the preceding claims, wherein a method of coating under vacuum is used in step a).

8. Apparatus for producing plane-parallel platelets, in particular for implementing the process according to any one of the preceding claims, comprising

a carrier (5) rotatable about an axis,

means (9a, 9b, 9c) for coating a partial surface of said carrier (5) with at least one product layer,

means (13) for stripping said product layer from said partial surface of said carrier in such a way that plane-parallel platelets are produced,

with transport of said partial surface between said coating means (9a, 9b, 9c) and said stripping means (13) being effected through rotation of said carrier (5).

9. The apparatus according to claim 8, wherein said carrier (5) is located in a vacuum chamber, and an intermediate separation (12a, 12b) for creating two pressure stages is provided between said coating means and said stripping means (13).

10. The apparatus according to claim 8 or 9, comprising means for coating said carrier with a separating agent layer prior to application of said product layer, wherein

said separating agent is an inorganic separating agent which may be evaporated in vacuum without dissociation,

said product layers include metals, oxides, fluorides or carbides, and

said carrier (5) comprises metal, glass, enamel, ceramic, or an organic material.

11. The apparatus according to any one of claims 7 to 10, wherein said carrier (5) comprises an open or closed, rotationally symmetrical, rigid body.

12. The apparatus according to any one of claims 7 to 10, wherein said carrier (5) comprises several open or closed, rotationally symmetrical, rigid bodies which rotate about a common axis or about several axes.

13. The apparatus according to claim 12, wherein said carrier (5) comprises several parallel discs of which at least one may be coated face-and-back by said coating means.

14. The process according to claim 1, wherein said partial surface of said carrier (5) is coated with an organic separating agent in step a) prior to application of said product layer, and said separating agent layer is melted in step c).

15. The process according to claim 14, wherein in step a) said partial surface of said carrier (5) is liquid-coated by dipping, rolling, pouring or spraying, in the further course of the rotating movement of said carrier (5) said separating agent layer solidifies on said carrier through cooling of said carrier, is subsequently vapor deposition coated with one or several product layers in high vacuum, and afterwards in step c) said separating agent layer is melted, wherein said product layer located thereon falls apart into flakes, to then be present as a mixture in said separating agent.

16. The apparatus according to claim 8 or 9, comprising means for coating said carrier with a separating agent layer prior to application of said product layer, wherein

said separating agent is a meltable organic separating agent,

said product layers include metals, oxides, fluorides or carbides, and

said carrier (5) comprises metal, glass, enamel, ceramic, or an organic material.